IS 354-3 (1986): Methods of sampling and test for resins for paints, Part 3: Special test methods for phenolic resins [CHD 20: Paints, Varnishes and Related Products]

BLANK PAGE


IS : 354 ( Part 3 ) - 1986
Reaffirmed - 2012

# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

## PART 3 SPECIAL TEST METHODS FOR PHENOLIC RESINS

## *( Second Revision )*

Second Reprint JANUARY 1996

UDC 677.621.633[ 678.652 ] : 620.1



**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

**Gr 2** *January* 1987

# AMENDMENT NO. 1 JANUARY 1990
TO
# IS : 354 ( Part 3 ) - 1986 METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS
## PART 3 SPECIAL TEST METHODS FOR PHENOLIC RESINS

## *( Second Revision )*

( *Page* 3, *clause* **0.2**, *line* 7 ) — Delete the words 'chlorinated rubber'.

( *Page* 3, *clause* **0.2**, *last line )* — Delete 'Part 8'.

( CDC 50 )

Printed at Printwell Printers, Delhi, India

# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

## PART 3 SPECIAL TEST METHODS FOR PHENOLIC RESINS

## *(Second Revision)*

Raw Materials for Paint Industry Sectional Committee, CDC 50

| *Chairman* | *Representing* |
|---|---|
| SHRI R. K. MARPHATIA | Goodlass Nerolac Paints Ltd, Bombay |
| *Members* | |
| SHRI S. S. ANAKAIKAR ( *Alternate* to Shri R. K. Marphatia ) | |
| SHRI K. M. BANERJEE | National Test House, Calcutta |
| SHRI S. BHATTACHARYYA | IEL Ltd, Calcutta |
| SHRI G. N. TIWARI ( *Alternate* ) | |
| CHEMIST & METALLURGIST-II | Ministry of Railways |
| ASSISTANT RESEARCH OFFICER ( CM-II ) ( *Alternate* ) | |
| SHRI M. C. CHOKSI | Resins & Plastics Pvt Ltd, Bombay |
| SHRI O. P. DHAMIJA | Export Inspection Council of India, Calcutta |
| SHRI S. N. DUTTA ( *Alternate* ) | |
| DR A. B. KARNIK | Colour-Chem Ltd, Bombay |
| SHRI M. E. MARATHE ( *Alternate* ) | |
| SHRI R. D. KAWATRA | Directorate General of Technical Development, New Delhi |
| SHRI KULTAR SINGH ( *Alternate* ) | |
| SHRI P. R. MALHAN | Development Commissioner ( SSI ) ( Ministry of Industry ), New Delhi |
| SHRI R. MUKOPADHYAY ( *Alternate* ) | |
| SHRI K. C. MEHTA | Indian Oil Corporation Ltd, Faridabad |
| SRHI B. RAMA MURTI | Travancore Titanium Products Ltd, Trivandrum |
| DR V. S. VAJAYAN NAYAR ( *Alternate* ) | |
| DR R. J. RATHI | Sudarshan Chemical Industries Ltd, Pune |
| SHRI K. L. RATHI ( *Alternate)* | |
| SHRI R. R. SEQUEIRA | Garware Paints Ltd, Bombay |
| SHRI C. R. THUSE ( *Alternate* ) | |

( *Continued on page* 2 )

( *Continued from page* 1 )

| *Members* | *Representing* |
|---|---|
| DR G. K. SINGHANIA | Ministry of Defence ( R & D ) |
| SHRI I. K. LOOMBA ( *Alternate* ) | |
| SHRI S. K. SRIVASTAVA | Oil Technologists' Association of India, Kanpur |
| SHRI SURENDRA GANG ( *Alternate* ) | |
| SHRI M. S. SULTANIA | Ministry of Defence ( DGI ) |
| SHRI R. S. SENGAR ( *Alternate* ) | |
| DR R. B. TIRODKAR | Asian Paints ( India ) Ltd, Bombay |
| SHRI V. M. NATU ( *Alternate* ) | |
| SHRI T. K. VISWANATHAN | Addisons Paints & Chemical Ltd, Madras |
| SHRI K. R. SANTHANAN ( *Alternate* ) | |
| SHRI P. M. VYAS | Indian Paint Association, Calcutta |
| SHRI D. K. ROY ( *Alternate* ) | |
| SHRI SATISH CHANDER, Director ( Chem ) | Director General, ISI ( *Ex-officio Member* ) |

*Secretary*

SHRI M. M. MALHOTRA
Deputy Director ( Chem ), ISI

## Resins Subcommittee, CDC 50 : 4

*Convener*

| | |
|---|---|
| SHRI M. C. CHOKSI | Resins & Plastics Pvt Ltd, Bombay |

*Members*

| | |
|---|---|
| ASSISTANT RESEARCH OFFICER ( CM-II ) | Ministry of Railways |
| SHRI S. K. ASTHANA | Ministry of Defence ( DGI ) |
| SHRI S. S. KATIYAR ( *Alternate* ) | |
| SHRI V. S. BHAKRE | Hindustan Ciba Geigy, Bombay |
| SHRI V. H. DESAI | ILAC India Ltd, Bombay |
| SHRI S. MITRA ( *Alternate* ) | |
| SHRI S. C. JAIN | Goodlass Nerolac Paints Ltd, Bombay |
| SHRI V. M. NATU | Asian Paints ( India ) Ltd, Bombay |
| DR A. V. RAO ( *Alternate* ) | |
| SHRI S. V. R. SEKHAR | Reichold Chemicals India Ltd, Madras |
| SHRI JANKIRAMAN (*Alternate*) | |
| SHRI G. N. TEWARI | IEL Limited, Calcutta |
| DR S. D. YADAV | Chowgule & Co ( Hind ) Pvt Ltd, Bombay |
| SHRI V. M. NACHANE ( *Alternate* ) | |

# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

## PART 3 SPECIAL TEST METHODS FOR PHENOLIC RESINS

## *( Second Revision )*

## 0. FOREWORD

**0.1** This Indian Standard ( Part 3 ) ( Second Revision ) was adopted by the Indian Standards Institution on 18 August 1986, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was originally published in 1952 covering methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for methods of sampling and test for natural and synthetic resins was published as Part 2 of the above standard in 1971. These two parts were amalgamated and revised in 1976. This revision has been necessitated as more and more newer synthetic resins like polyamides, polyvinyls, chlorinated rubber and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee felt it appropriate to publish this standard in various parts, as indicated below:

Part 1 General test methods
Part 2 Special test methods for alkyd resins
Part 3 Special test methods for phenolic resins
Part 4 Special test methods for epoxy resins
Part 5 Special test methods for polyamide resins
Part 6 Special test methods for amino resins
Part 7 Special test methods for determination of monomer content in acrylic or vinylacetate containing polymers and emulsions
Part 8 Special test methods for chlorinated rubber

**0.3** In this standard ( Part 3 ), test methods covered in **18.1** and **18.2** of IS : 354-1976* have been included. In addition to above, methods of test for determination of methylol content and reactivity with tung oil have been added.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

## 1. SCOPE

**1.1** This standard ( Part 3 ) prescribes the special test methods for phenolic resins used in paints and enamels.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard ( Part 3 ), the definitions given in IS : 1303-1963: and IS : 6667-1972§ shall apply.

## 3. SAMPLING

**3.1** Representative samples of the phenolic resins shall be drawn as prescribed in **3** of Part 1 of this standard.

## 4. IDENTIFICATION

**4.1 Phenolphthalein Test** — Heat 1 g of resin with about 1 g of phthalic anhydride and 3 drops of concentrated sulphuric acid until a rich brown melt develops, cool and dilute with water and make alkaline with 10 percent sodium hydroxide solution. Characteristic red colour of phenolphthalein indicates presence of phenols. In case tarry matter obscures colour, dilute with water and confirm by discharging colour by acids. All phenolics except the oil-modified ones, give a positive reaction with this test.

**4.2 Millon Test** — Dissolve 10 g of mercury in 10 g of fuming nitric acid without heating. Dilute with twice its volume of water and filter off any precipitate or allow it to settle. Heat a small piece of resin with one millilitre of the above clear reagent and boil for about 2 minutes. A red colour indicates phenol. This test is responded by some proteins also, but absence of nitrogen confirms phenols. A few para-substituted phenolics fail to yield a positive test.

*Methods of sampling and test for resins for paints *( first revision )*.
†Rules for rounding off numerical values ( *revised* ).
‡Glossary of terms relating to paints ( *second revision* ).
§Glossary of terms used in synthetic resin industry.

## 5. DETERMINATION OF FREE PHENOLS

**5.0 Outline of the Methods** — These methods are applicable to all phenolic resins except those containing *p*-phenyl phenol. Method A applies to simple phenols up to and including xylenols and Method B to common alkylated phenols. In Method A, free phenols are isolated by steam distillation, reacted with a measured excess of bromine, and the excess back titrated with standard sodium thiosulphate solution. Method B is similar to Method A except that it employs iodine for reaction.

### 5.1 Apparatus

**5.1.1** *Steam Generating Source*

**5.1.2** *Distillation Flask*

### 5.2 Reagents

**5.2.1** *Sodium Hydroxide Solution* — Dissolve 100 g of sodium hydroxide in water and dilute to 1 000 ml.

**5.2.2** *Bromide-Bromate Solution* — Dissolve 2.784 g of potassium bromate and 10 g of potassium bromide in 1 000 ml of water.

**5.2.3** *Potassium Iodide Solution* — Dissolve 100 g of potassium iodide in water and dilute to 1 000 ml.

**5.2.4** *Standard Sodium Thiosulphate Solution* — 0.1 N.

**5.2.5** *Iodine Solution* — Dissolve 4.2 g of iodine in 15 g of a saturated aqueous potassium iodide solution and dilute to one litre.

**5.2.6** *Sulphuric Acid* — dilute, 1:19 ( *v/v* ).

**5.2.7** *Sodium Bicarbonate Solution* — Dissolve 84 g of sodium bicarbonate in water and dilute to 1 000 ml.

### 5.3 Procedure

**5.3.1** *Isolation of Phenols* — Weigh accurately 1 to 2 g of the material and transfer to a long-necked flask. Add 50 ml of water and set the distillation assembly. Pass steam from the steam generator through the contents and collect the distillate in a 1 000 ml volumetric flask as receiver. Collect nearly 900 ml of distillate. Apply, if necessary, a small flame to the bottom of the long-necked flask during distillation to maintain the volume of water constant. If the distillate is clear, make up to mark; if not clear, add a few millilitres of sodium hydroxide solution to dissolve the insoluble phenols.

**5.3.2** *Method A* — Pipette 25 ml of the distillate into a 500-ml iodine flask. Add 25 ml of potassium bromide-bromate solution, shake and add 10 ml of hydrochloric acid ( *see* Note ). Stopper the flask, shake to mix well and add a little water to the neck junction of stopper and flask. Let it stand for 15 minutes.

Remove the stopper carefully, add 10 ml of potassium iodide solution, shake and wash down the stopper and walls of the flask. Titrate the solution against standard sodium thiosulphate using starch solution as indicator. Conduct a blank simultaneously using all the reagents except the sample.

NOTE — If the bromine colour disappears during shaking, take a fresh aliquot, add double the amount of bromide-bromate solution.

Calculate the percentage of phenol as follows:

$$\text{Phenols, percent by mass} = \frac{(V_1 - V_2) \times N \times 1{\cdot}567}{M}$$

where

$V_1$ = volume in ml of standard sodium thiosulphate solution required for titration of blank,

$V_2$ = volume in ml of standard sodium thiosulphate solution required for titration of the sample,

$N$ = normality of standard sodium thiosulphate solution, and

$M$ = mass in g of the sample present in the aliquot used.

**5.3.3** *Method B* — Carry out the distillation as prescribed in **5.3.2.** Add 15 ml of sodium hydroxide solution to the distillate and dilute to one litre. Add 100 ml of water to a 500-ml iodine flask. Pipette 10 ml of the solution into the flask and add 30 ml of the iodine solution. Stopper and shake well for 5 minutes. Add 50 ml of sulphuric acid to the flask and titrate with 0.1 N sodium thiosulphate solution to a colourless end point (*see* Note), using one millilitre of starch solution as indicator. Carry out a blank simultaneously using all the reagents but without the material.

Calculate the percentage of phenol as follows:

$$\text{Phenols, percent by mass} = \frac{(V_1 - V_2) \times N \times F}{M}$$

where

$V_1$ = volume in ml of standard sodium thiosulphate required in blank determination;

$V_2$ = volume in ml of standard sodium thiosulphate required in the determination with the sample;

$N$ = normality of standard sodium thiosulphate solution;

$F$ = conversion factor, 3.755, 4.106 and 4.255 for *p*-tertiary butyl phenol, *p-tertiary* amyl phenol and *p*-tertiary phenyl phenol respectively; and

$M$ = mass in g of the material present in the aliquot taken for determination.

NOTE — If the material it known to be pure *p*-phenyl phenol, add 50 ml of the sodium bicarbonate solution to the flask before the sample is added to prevent the formation of colour that interferes with the titration end point.

## 6. DETERMINATION OF METHYLOL CONTENT

**6.0 Outline of the Method** — The method involves reaction of the resin with phenol and from the amount of water of reaction, the methylol content is calculated.

### 6.1 Apparatus

**6.1.1** *Dean and Stark Apparatus* — consisting of 1 000-ml capacity round flask, double surface condenser, spray tube and a receiver.

### 6.2 Reagents

**6.2.1** *Phinol — see* IS : 538-1968*.

**6.2.2** *Toluene — see* IS : 537-1967†.

**6.2.3** *Paratoluem Sulphonic Acid*

### 6.3 Procedure

**6.3.1** *Sample Reading* — Take 100 g of phenol, 300 ml of dry toluene and 15 g of paratoluene sulphonic acid into the round bottom flask. Attach the flask to the Dean and Stark condensing and collecting system and heat the flask over a mantle at such a rate that the condensate falls from the end of the condenser at a rate of two to five drops per second. Continue the distillation until condensed water, if any, is no longer visible in any part of the apparatus except at the bottom of the graduated tube, cool the flask to room temperature and remove, if any, water collected in the graduated tube. Weigh accurately around 50 g of a powdered resin sample directly into the phenol in the round bottom flask without sticking to the sides of the ground joint. Refit Dean and Stark condensing and collecting system. Heat to reflux and continue till no more water is evolved. Measure the water liberated accurately ( sample reading ).

*Specification for phenol ( carbolic acid ) ( *first revision* ).
†Specification for toluene, pure nitration grade ( *first revision* ).

**6.3.2** *Blank Reading* — Repeat the procedure given in **6.3.1** taking 300 ml toluene and 15 g of paratoluene sulphonic acid. Remove, if any, water collected in graduation tube and adding accurately around 50 g of powdered resin sample. Measure the water liberated accurately ( blank reading ).

**6.4 Calculation**

$$\text{Methylol content in percentage} = \frac{(V_2 - V_1) \times 31 \times 100}{18 \times M}$$

where

$V_2$ = volume in ml of water collected for sample,

$V_1$ = volume in ml of water collected for blank, and

$M$ = mass in g of the material taken for test in **6.3.**

## 7. REACTIVITY WITH TUNG OIL

**7.0 Outline of the Method** — Phenolic resins react with tung oil at temperature about 180°C and produce products of increasing viscosity with increase in time of heating, to gel at the end.

**7.1 Procedure** — Fix up 400-ml beaker with a mechanical stirrer over a heat source. Fill three-fourths of the beaker with paraffin oil. Fix a thermometer so that its bulb is close to sample in the test tube when the latter is fixed. Start the heat source and stirrer. Maintain the temperature of the paraffin oil at 180 ± 1°C.

Weigh 5 g of tung oil along with 5 g of powdered phenolic resin in the test tube having a capacity of 25 ml and fix the test tube with the phenolic resin oil mixture dipping below paraffin oil layer in the beaker. Insert 10 mm diameter glass rod inside the test tube. Start stop-watch immediately and with slow swirling, continue the heating of the mixture in the test tube at 180 ± 1°C. When there is resistance to swirling, stop the watch and note the time. This denotes the gel time.

# BUREAU OF INDIAN STANDARDS


*Headquarters* :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 331 01 31
331 13 75

Telegrams : Manaksanatha
(Common to all Offices)

| *Regional Offices* : | *Telephone* |
|---|---|
| Central : Manak Bhavan, 9, Bahadur Shah Zafar Marg, NEW DELHI 110002 | { 331 01 31<br>{ 331 13 75 |
| * Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054 | 37 86 62 |
| Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036 | 2 18 43 |
| Southern : C.I.T. Campus, IV Cross Road, MADRAS 600113 | 41 29 16 |
| † Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093 | 6 32 92 95 |
| *Branch Offices* : | |
| 'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001 | 2 63 48 |
| ‡ Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058 | 39 49 55 |
| Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003 | 55 40 21 |
| Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002 | 5 36 27 |
| Kalai Kathir Building, 6/48-A Avanasi Road, COIMBATORE 641037 | 2 67 05 |
| Quality Marking Centre, N.H. IV, N.I.T., FARIDABAD 121001 | – |
| Savitri Complex, 116 G. T. Road, GHAZIABAD 201001 | 8-71 19 96 |
| 53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 | 3 31 77 |
| 5-8-56C L. N. Gupta Marg, ( Nampally Station Road ) HYDERABAD 500001 | 23 10 83 |
| R14 Yudhister Marg, C Scheme, JAIPUR 302005 | 6 34 71 |
| 117/418 B Sarvodaya Nagar, KANPUR 208005 | 21 68 76 |
| Plot No. A-9, House No. 561/63, Sindhu Nagar, Kanpur Road, LUCKNOW 226005 | 5 55 07 |
| Patliputra Industrial Estate, PATNA 800013 | 6 23 05 |
| District Industries Centre Complex, Bagh-e-Ali Maiden, SRINAGAR 190011 | |
| T. C. No. 14/1421, University P.O., Palayam, THIRUVANANTHAPURAM 695034 | 6 21 04 |
| *Inspection Offices* (With Sale Point) : | |
| Pushpanjali, First Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010 | 52 51 71 |
| Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005 | 5 24 35 |
| *Sales Office Calcutta is at 5 Chowringhee Approach, P. O. Princep Street, CALCUTTA | 27 68 00 |
| † Sales Office is at Novelty Chambers, Grant Road, BOMBAY | 89 65 28 |
| ‡ Sales Office is at Unity Building, Narasimharaja Square, BANGALORE | 22 39 71 |



Reprography Unit, BIS, New Delhi, India